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Meet Our New Colleagues

This column presents selected currently graduating Ph.D. students in the thermal spray field from around the world. Students planning to graduate in the area of thermal spray within next three to six months are encouraged to submit a short description (one to two pages, preferably as Word document) of the projects they performed during their studies to Jan Ilavsky, JTST associate editor, address: Argonne National Laboratory, Advanced Photon Source, 9700 S. Cass Ave., Argonne, IL, 60439; e-mail: JTST .Ilavsky@aps.anl.gov. After limited review and corrections and with agreement of the student's thesis advisor, selected submissions will be published in the upcoming issues of JTST.

Influence of Geometrical and Spatial Characteristics of the Porosity on the Thermal Conductivity of EB-PVD TBCs

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Metallic blades

working at the

high-temperature

sector (after the

combustion cham-

ber) of aircraft and

stationary turbines

are nowadays

protected against

heat, corrosion,

oxidation, and

erosion by thermal



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barrier coatings (TBCs) made via electron-beam physical vapor deposition (EB-PVD) or air plasma spray (APS). To fulfill all these functions, TBCs are composed by several components: ceramic top coat (TC), thermal grown oxide (TGO), and metallic bond coat (BC). One of the principal key physical properties of the top coat is the thermal isolation capability, which is directly related to the chemical composition and microstructure of the deposited ceramic (Ref 1-3). Moreover, the influence of the microstructure on the thermal conductivity of EB-PVD TBCs is specifically related to the spatial and geometrical characteristics of their porosity (Ref 1, 4, 5). Thus, a viable and profitable optimization of the thermal isolation effectiveness of ceramic top coats could be achieved by understanding the relation: thermal conductivity/microstructure. With this work, we seek to develop basic knowledge of this significant relationship. Further, we also expect to develop understanding of the correlation between the values of the EB-PVD manufacturing parameters and the resulting microstructure, gaining insight into the physics of the process itself.

EB-PVD TBCs exhibit a microstructure composed of parallel conical columns grown in a direction perpendicular to the plane of the substrate separated by intercolumnar gaps. Additionally, featherarms gaps form inside of each column grown until they reach the periphery, increasing the open porosity together with the intercolumnar gaps. Finally, arrays of closed intracolumnar pores form inside of each column after every rotation of the substrates during the coating process. Due to the differences in size, accessibility, and anisotropic shapes and orientation of the pores, the complete analysis of their geometrical and spatial characteristics could only be achieved using a sophisticated measuring technique/equipment such as ultrasmall-angle x-ray scattering (USAXS) (Ref 6). In the present work, we characterize three different morphologies in as-coated and after 1100 °C/100 h heat treatment conditions via the 2D-collimated USAXS instrument at the UNICAT beam line 33-ID, Advanced Photon Source, ANL (Ref 7). Samples were manufactured by varying the EB-PVD process parameters, namely substrate temperature and rotations speed (see Fig. 1). The obtained USAXS data were related via computer-based modeling to values of geometrical and spatial characteristics of the pore populations, namely: volume, shape, size, aspect ratio, and orientation; which represent statistical average values based on Gaussian distributions (Ref 8). In addition, thermal conductivity measurements of the corresponding microstructures were done via laser flash method (LFA) employing a Netsch-LFA 427 equipment.

Figure 2 shows the experimental and modeled polar distribution graphs of USAXS-scattering intensities versus azimuthal angle (α), corresponding to the "feathery" microstructure in as-coated conditions manufactured at low substrate temperature/fast rotation speed. According to the results of the modeling, the three microstructures have similar dimensions and volume at their intercolumnar pores. The "feathery" microstructure contains slightly larger feather-arms pores followed by the "coarse" and "intermediate." However, the noticeable difference between the coatings resides in the size and volume of intracolumnar pores. At the "feathery" microstructure, these pores are the thinnest and contain evidently higher volume values compared with the "intermediate" and "coarse" microstructures, respectively. The relation between the intracolumnar pores volume of the three coatings agrees with that of the val-



Fig. 1 Scanning electron micrographs of EB-PVD TBCs cross sections manufactured at different temperatures and rotation speeds conditions. (a) "Intermediate," 950 °C/12 rpm. (b) "Coarse," 1000 °C/3 rpm. (c) "Feathery," 850 °C/30 rpm



Fig. 2 Polar distribution of the USAXS-scattering intensities measured at different q values versus azimuthal angle (α) corresponding to the "feathery" EB-PVD TBCs microstructure



Fig. 3 Thermal conductivity versus measuring temperature values of three EB-PVD TBCs produced by varying the process parameters: coarse, intermediate, and feathery

ues of their thermal conductivity as shown in Fig. 3. Additionally, after the heat treatment of the coatings at 1100 °C/ 100 h, an increase of the thermal conductivity at all microstructures occurs. Still, the "feathery" structure contains the better distribution of shaped intracolumnar spherical pores due to the higher content of pores arrays. Also, according to the USAXS-modeling, this microstructure retains its dimensions of the feather-arms, which also obstruct the heat (phonons) transfer through the columns of the coating.

In conclusion, the thermal conductivity of EB-PVD TBCs can be influenced via controlling of the process parameters during the coating process. Specifically, low substrate temperatures and high rotation speed create the thermophysical conditions for the formation and continuous growth of an elevated number of intracolumnar pores along the complete width of the columns until they reach the periphery, creating additionally deeper and stable gaps between feather-arms. Modeling of the thermal conductivity based on the spatial and geometrical characteristics of all pores at EB-PVD TBCs is under development.

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References

- D.D. Hass, A.J. Slifka, and H.N.G. Wadley, Low Thermal Conductivity Vapor Deposited Zirconia Microstructures, *Acta Mater.*, 2001, 49, p 973-983
- J.R. Nicholls et al., Methods to Reduce the Thermal Conductivity of EB-PVD TBCs, *Surf. Coat. Technol.*, 2002, 151-152, p 383-391
- C.G. Levi, Emerging Materials and Processes for Thermal Barrier Coatings, *Current Opinion Solid State Mater. Sci.*, 2004, 8, p 77-91
- S. Gu et al., Thermal Conductivity of Zirconia Coatings with Zig-Zag Pore Microstructures, *Acta Mater.*, 2001, 49(13), 2001, p 2539-2547
- P. Heydt, C. Lou, and D.R. Clarke, Crystallographic Texture and Thermal Conductivity of Zirconia Thermal Barrier Coatings Deposited on Different Substrates, *J. Am. Ceram. Soc.*, 2001, 84(7), 2001, p 1539-1544
- A.J. Allen, Characterization of Ceramics by X-Ray and Neutron Small-Angle Scattering, J. Am. Ceram. Soc., 2005, 88(6), p 1367-1381
- J. Ilavsky et al., Effective Pinhole-Collimated Ultrasmall-Angle X-Ray Scattering Instrument for Measuring Anisotropic Microstructures, *Rev. Sci. Instrum.*, 2002, 73(3), p 1-3
- J. Ilavsky et al., Microstructure Characterization of Thermal Barrier Coatings Deposits— Practical Models from Measurements, in International Thermal Spray Conference, Basel, Switzerland, 2005